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SYNTHESIS OF ENERGETIC MATERIALS(U) NAVAL SURFACE  
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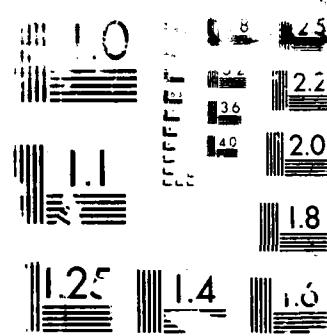
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SYNTHESIS OF ENERGETIC MATERIALS  
ANNUAL PROGRESS REPORT FOR THE  
OFFICE OF NAVAL RESEARCH

WORK REQUEST NO00187WX24109

M. CHAYKOVSKY  
W. M. KOPPES

MARCH 1987

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RESEARCH AND TECHNOLOGY DEPARTMENT

NAVAL SURFACE WARFARE CENTER  
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19. ABSTRACT (Continue on reverse if necessary and identify by block number) The chemistry of the 2,6-diazabicyclooctane ring system was studied toward the synthesis of tetra- and hexa-substituted derivatives. Stable dinitro derivatives were prepared but attempts to prepare tetranitro derivatives by oxidative nitration were unsuccessful, and work on the diazabicyclooctanes was terminated. Methylene dinitramine (MEDINA) was successfully condensed with glyoxal in acetic anhydride to give a substitute dinitroimidazolidine which shows promise as a precursor for the synthesis of bicyclo-HMX. Trichloroacetamidine condensed with glyoxal and with oxalyl chloride to give a tri-chloromethyl substituted dihydroxyimidazoline and imidazolinedione respectively. The imidazoline was unstable and could not be converted into the bistrichloromethyl substituted tetraazabicyclooctane ring system by further condensation with trichloroacetamidine. The stable imidazolinedione, on the other hand, is a potential intermediate for such a condensation reaction. Trihalomethyl-bisformamides and bisacetamides are also potential precursors for this substituted bicyclic ring system. The trihalomethyl groups should stabilize the ring during nitrolysis reactions leading to the bicyclo-HMX system.					
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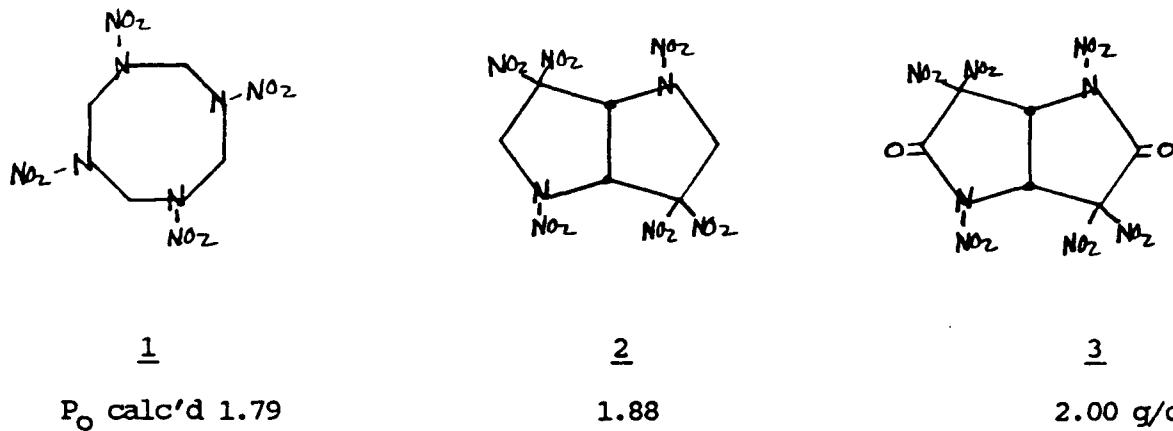
## INTRODUCTION

The work described in this report was carried out during 1986 under the sponsorship of the Office of Naval Research, Code 1132P (Dr. R. S. Miller). The effort consisted of two separate tasks: (1) synthesis of energetic monomers and polymers, and (2) synthesis of polycyclic and adamantoid nitramines. Task (1) will be reported on under separate cover by Dr. H. G. Adolph and collaborators. Task (2) is covered in this report. The principal objective of the work is the syntheses of nitramines with high crystal density and energy-density greater than that of HMX.

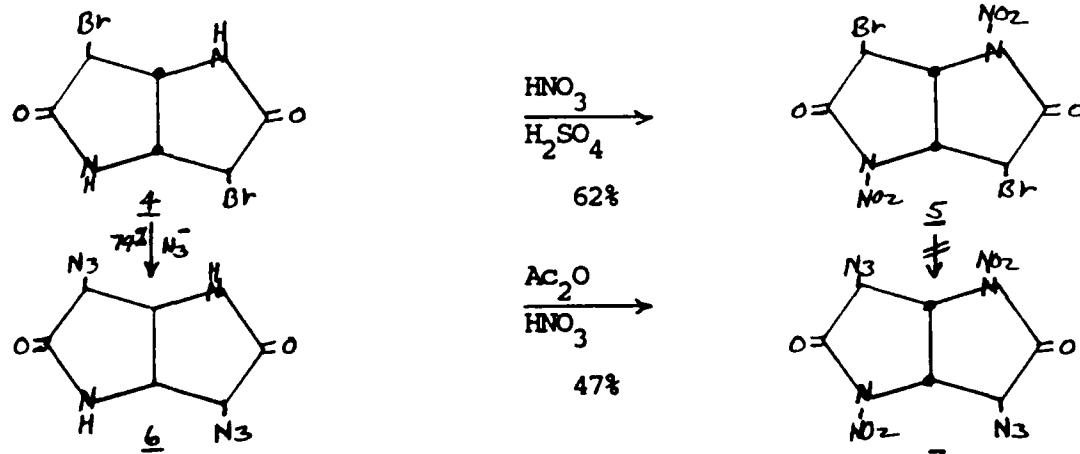
SYNTHESIS OF POTENTIALLY DENSE NITRAMINES

(a) Diazabicyclooctanes.

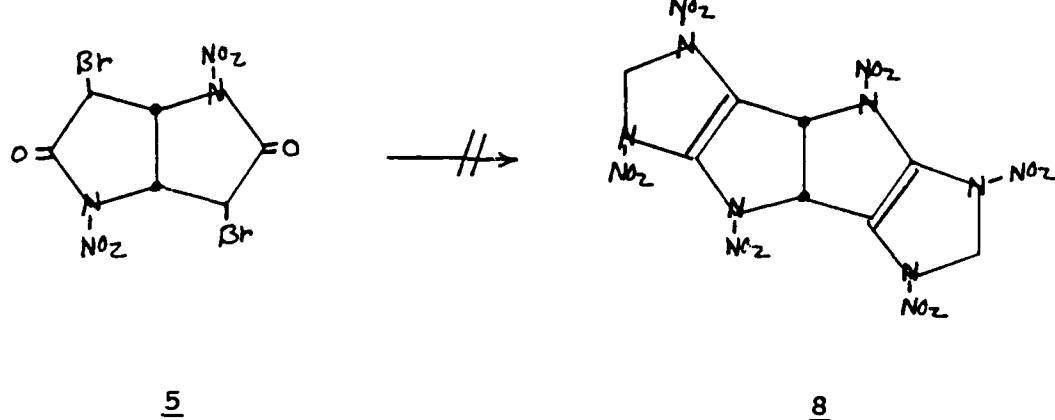
During the past year, work was continued on the synthesis of the diazabicyclooctanes 2 and 3. These compounds have calculated densities significantly higher than that of HMX (1) and estimated detonation pressures in the range of 420-430 kbar, about 10% greater than that of HMX.



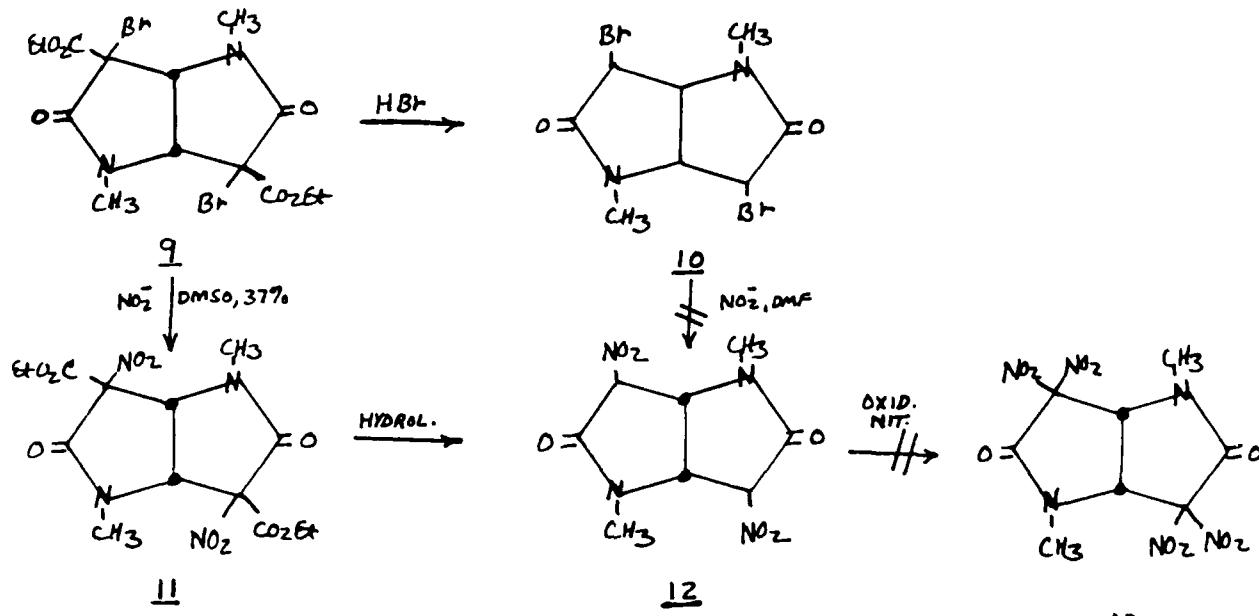
In our last report<sup>1</sup>, we described the synthesis of the bicyclic dibromide (4). Attempts were made to displace the bromines in 4 with nitrite ion in a typical Kornblum reaction<sup>2</sup>. However, the corresponding dinitro compound could not be obtained. Dibromide (4) turned out to be completely inert under various reaction conditions, probably as a result of the exo-nature of the bromines on the cis-fused ring system, which precludes backside attack by nitrite ion. Mixed acid nitration converted the dibromide (4) into the cyclic dinitramide (5). This compound was extremely susceptible to ring-opening reactions upon treatment with nucleophilic reagents. Reaction of 5 with sodium nitrite or lithium azide gave only dark water-soluble products. On the other hand, reaction of lithium azide with 4 gave the diazide (6) in good yield, which could be nitrated to the diazido dinitramide (7).



We attempted to utilize compounds 5 and 7 as starting materials for the synthesis of some energetic tetracyclic compounds. For example, reaction of 5 with methylenedinitramine (MEDINA) under suitable reaction conditions might be expected to yield the tetracyclic 8. However, this and other attempted reactions on 5 and 7 were unsuccessful.



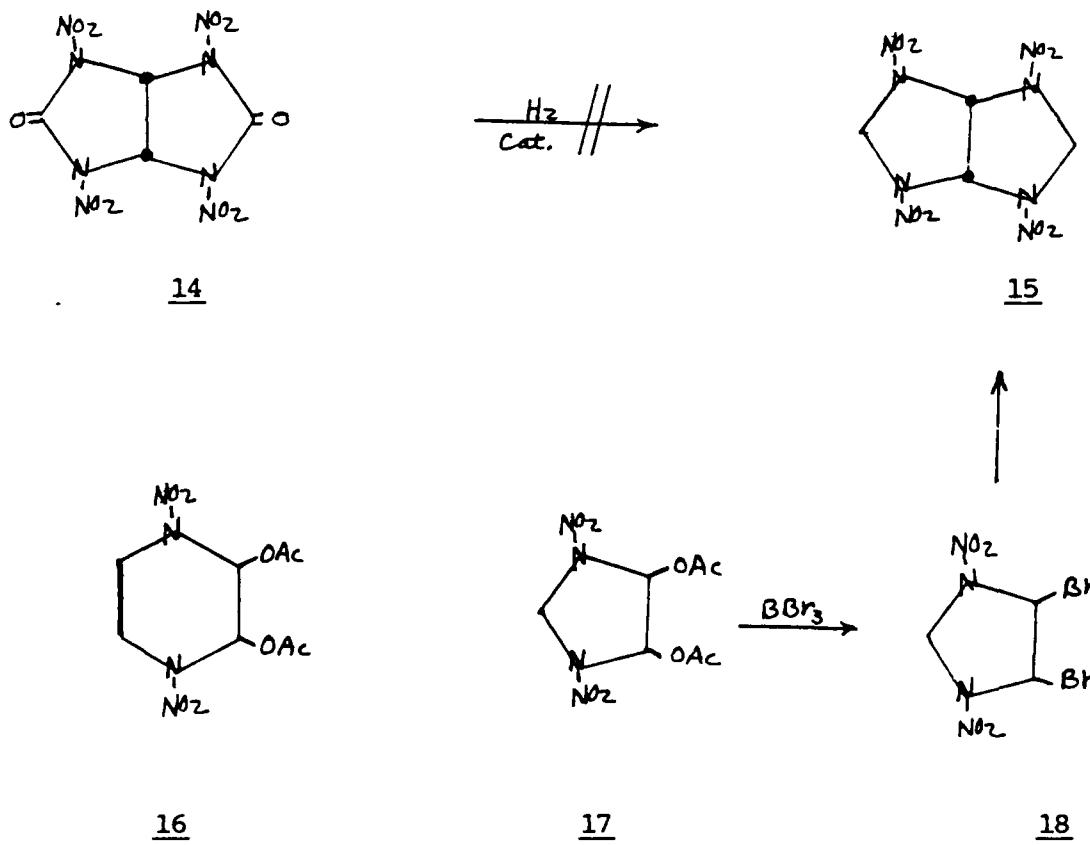
In another approach to the target diazabicyclooctanes 2 and 3, the dibromodiester (9) was prepared by bromination of the parent diester<sup>1</sup>. Hydrolysis gave the dibromide (10). This compound, similarly to 4, was also inert toward nitration under Kornblum conditions. However, the dibromodiester (9) was converted, by nitrite ion in DMSO, into the dinitrodiester (11) in moderate yield<sup>3</sup>. Basic hydrolysis then gave the dinitro bicyclic amide (12). Many attempts were then made to convert 12 into the tetrapnitro intermediate (13) using various oxidative nitration techniques<sup>4,5</sup>. Since none of these attempts were successful, synthetic work on these diazabicyclooctanes was terminated.



(b) Bicyclo HMX and Derivatives

During this period, work was continued on the chemistry of cyclic nitramines with the goal of synthesizing bicyclo-HMX and derivatives. Since tetrinitroglycouril (TNGU, 14) is a readily available compound, attempts were made to catalytically hydrogenolyze the carbonyl group under mild conditions of temperature and pressure to obtain bicyclo-HMX (15). Various catalysts such as platinum or palladium on carbon, and Lindlar's catalyst ( $Pd-CaCO_3-Pb$ ) were used. In all cases, complex mixtures of solid amorphous products were obtained from which no single crystalline compound could be separated. The NMR spectra of these mixtures did not show signal peaks which could be expected from bicyclo-HMX.

Several years ago<sup>6</sup>, we successfully condensed ethylenedinitramine (EDNA) with glyoxal in acetic anhydride to give the cyclic dinitramine (16). The corresponding reaction with methylenedinitramine (MEDINA) proved to be extremely difficult because of polymer formation. However, we now have conditions for the synthesis of the 5-membered cyclic dinitramine (17) in low yield. Following reaction schemes worked out on 16, it should be possible to obtain the dibromide (18), which is a precursor for the synthesis of bicyclo-HMX.

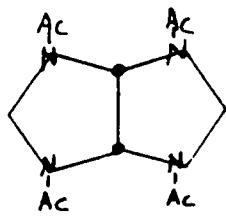


In another approach to the bicyclo-HMX structure, the reactions of amidines with glyoxal and oxalyl chloride were investigated. The strategy was to synthesize the tetraazabicyclooctane ring system with groups at the 3,7-positions that would stabilize the ring system toward nitration, as was done successfully in the synthesis of the tetrakis-trifluoromethyl bicyclo-HMX.<sup>1</sup> The amidines, however, would lead to structures with only two pendant trihalomethyl groups on the bicyclo-HMX (Scheme 1). The trichloromethyl groups could potentially be replaced by hydrogen.

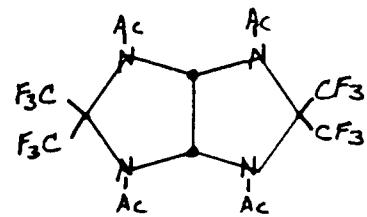
The trichloro- and trifluoroacetamidines were easily prepared by reaction of the corresponding nitriles with ammonia. Attempts to prepare a tetraazabicyclooctane (20) from trichloroacetamidine and aqueous glyoxal were unsuccessful over a wide range of pH values. In an ethanol-water system, the amidine-glyoxal addition product (19) was obtained. This adduct, which appears to be the first of its type observed, is a labile compound which turned dark on storage at room temperature, but which gave a molecular ion in the mass spectral analysis. Its further reaction with trichloroacetamidine to give 20 was attempted in various solvents under acid catalysis. In all cases, the solutions turned dark due to the decomposition of 19, and no products could be isolated. Attempts to convert 19 to its diacetate derivative, which should also be capable of condensation with the amidine, were also unsuccessful due to extensive decomposition.

In order to obtain a more stable reaction intermediate than 19, trichloroacetamidine was condensed with oxalyl chloride to give the dione (21), isolated as the hydrochloride salt. Experiments are underway to condense this compound with trichloroacetamidine to give the bicyclic product (22), in analogy to a similar reaction conducted with benzamidine.<sup>7</sup>

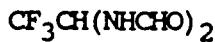
We have previously reported the synthesis of the tetraazabicyclic compound (24) by stepwise condensation of methylenebisacetamide with glyoxal.<sup>6,8</sup> Similar reactions are now underway aimed at the synthesis of trihalomethyl substituted bicyclic systems such as 25, starting with the known trifluoromethyl<sup>9</sup> and trichloromethyl<sup>10</sup> substituted bisamides 26 and 27.



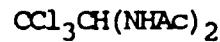
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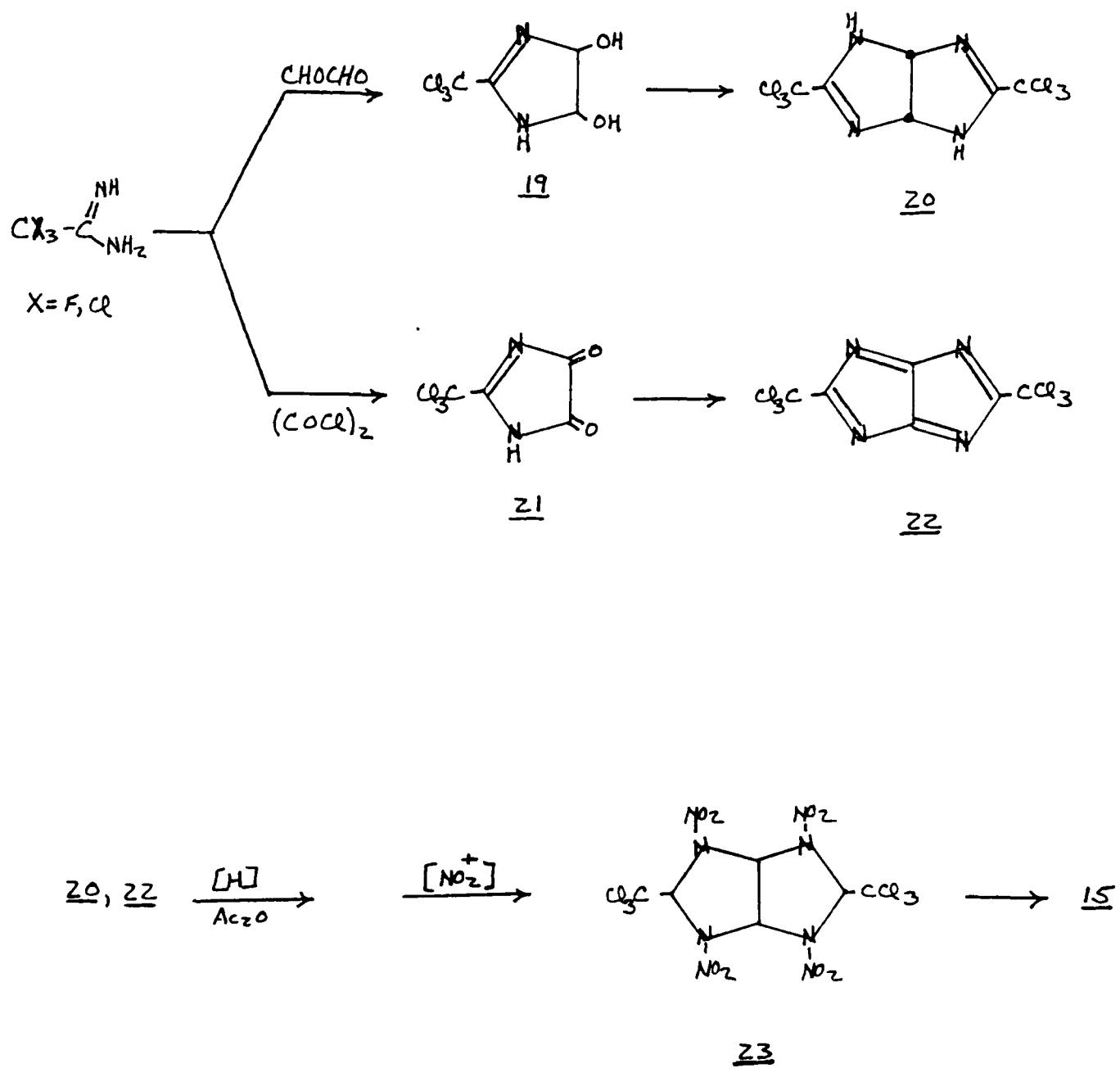


26



27

Scheme 1



## EXPERIMENTAL SECTION

Melting points are uncorrected. Temperatures are in °C. Microanalyses are by Galbraith Laboratories, Knoxville, Tennessee. NMR spectra were obtained in part on a Varian EM-390 spectrometer, in part on a Varian XL-200 NMR spectrometer. Chemical shifts are in ppm relative to TMS internal standard.

4,8-Diazido-3,7-dioxo-2,6-diazabicyclo[3.3.0]octane(6). - A solution of 4 (2.98g, 10 mmol) and lithium azide (4.90g, 100 mmol) in DMSO (25mL) was stirred at room temperature for 26 h, then poured into a mixture of ice and water (125mL). After stirring for 30 min in an ice bath, the precipitate was filtered to yield 1.50g of white solid: mp 208-210°C (dec). Cooling the filtrate overnight in a refrigerator gave an additional 0.145g of solid. The total yield was 74%. Recrystallization from ethanol gave colorless needles: mp 220-210°C (dec); IR (KBr), 2280 (w, sh), 2240 (w, sh), 2135 (s, N<sub>3</sub>), 2115 (s, N<sub>3</sub>), 1710 cm<sup>-1</sup> (s, CO); <sup>1</sup>H NMR (CF<sub>3</sub>CO<sub>2</sub>H) δ 8.33 (br s, 2H, NH), 4.63 (d, 4H, CHN<sub>3</sub> and CHN) ppm; mass spectrum (CI, CH<sub>4</sub>) m/z 263 (M+41, 0.9), 251 (M+29, 8.6), 223 (M+1, 18.7), 97 (100). Anal. Calcd. for C<sub>6</sub>H<sub>6</sub>N<sub>8</sub>O<sub>2</sub>: C, 32.43; H, 2.72; N, 50.44. Found: C, 32.18; H, 2.89; N, 49.26.

4,8-Diazido-2,6-dinitro-3,7-dioxo-2,6-diazabicyclo[3.3.0]octane(7). - Acetic anhydride (1.0 mL) was cooled to 0°C and absolute (98%) nitric acid (1.0 mL) was then added dropwise, with stirring, over 2 min. After 20 min, 6 (666mg, 3.0 mmol) was added all at once and stirring was continued at 0°C for 2h. The solution was then poured into a mixture of ice cold water (15 mL) and the precipitate filtered immediately and dried over P<sub>2</sub>O<sub>5</sub> under vacuum to yield 440mg (47%) of white solid: mp 123-124°C (dec); IR (KBr), 2140 (s), 1782 (s), 1590 (s), 1265 (s), 1158cm<sup>-1</sup> (s); <sup>1</sup>H NMR (Me<sub>2</sub>CO-d<sub>6</sub>) δ 5.42 (s, 4H, CHN<sub>3</sub> and CHN) ppm; mass spectrum (CI, CH<sub>4</sub>) m/z 353 (M+41, 0.1), 341 (M+29, 0.2), 313 (M+1, 0.55), 95 (50), 48 (48), 44 (100).

4,8-Dibromo-2,6-dimethyl-3,7-dioxo-2,6-diazabicyclo[3.3.0]octane(10). - A mixture of 9 (1.41g, 3.0 mmol) and aqueous HBr (15 mL of 5N) was refluxed for 3 h and the resulting solution was then evaporated under vacuum. The yellow residue was dissolved in a mixture of acetone (10 mL) and ether (3 mL) and cooled in a refrigerator overnight to deposit 660mg (67.5%) of a white solid: mp 216-218°C (dec). Recrystallization from ethanol gave colorless crystals: mp 220-222°C (dec); <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 3.01 (s, 6H, CH<sub>3</sub>), 4.41 and 4.52 (2s, 4H, CHBr and CHN) ppm; Anal. Calcd. for C<sub>8</sub>H<sub>10</sub>Br<sub>2</sub>N<sub>2</sub>O<sub>2</sub>: C, 29.47; H, 3.09; N, 8.59; Br, 49.03. Found: C, 29.76; H, 3.04; N, 8.58; Br, 47.02.

2,6-Dimethyl-4,8-dinitro-3,7-dioxo-2,6-diazabicyclo[3.3.0]octane(12). - A mixture of 11 (3.6g, 8.96 mmol) and 2N NaOH (22 mL) was stirred in an ice bath for 30 min and then with the bath removed for 90 min. The solution was again cooled in ice during the dropwise addition of 12N HCl (5 mL) over 5 min. After stirring at 0°C for 20 min, the mixture was filtered to yield 1.44g (62.3%) of white solid: mp 212-215°C (dec). Recrystallization from CH<sub>3</sub>CN gave colorless crystals: mp 239-240°C (dec); <sup>1</sup>H NMR (CF<sub>3</sub>CO<sub>2</sub>H) δ 5.81 (m, 2H, CHNO<sub>2</sub>), 5.39 (m, 2H, CHN), 3.31 (s, 6H, CH<sub>3</sub>) ppm; mass spectrum (CI, CH<sub>4</sub>) m/z 299 (M+41, 4.5), 287 (M+29, 5.6), 259 (M+1, 100); Anal. Calcd. for C<sub>8</sub>H<sub>10</sub>N<sub>4</sub>O<sub>6</sub>: C, 37.21; H, 3.90; N, 21.70. Found: C, 37.38; H, 3.98; N, 21.64.

4,5-Diacetoxy-1,3-Dinitroimidazolidine(17). - Concentrated H<sub>2</sub>SO<sub>4</sub> (0.3 mL) was

slowly added dropwise to a stirred mixture of methylenedinitramine (1.36g, 10 mmol), 40% aqueous glyoxal (1.6g, 11 mmol) and water (0.5 mL). The mixture was then slowly heated to 50°C to effect solution and acetic anhydride (15 mL) was added dropwise over 10 min. After stirring at room temperature for 18h, the cloudy mixture was evaporated under vacuum to about 3 mL and extracted thoroughly with ethyl acetate. The extracts were washed with H<sub>2</sub>O, 5% aqueous NaHCO<sub>3</sub>, saturated salt solution, dried (MgSO<sub>4</sub>) and evaporated to a pale yellow oil. The oil was dissolved in a minimum amount of warm 5% CH<sub>3</sub>CN-benzene, placed on a short column of silica gel (20g) and eluted with the same solvent mixture. The first 40 mL of eluent contained the desired product.

Evaporation gave a mixture of solid and oil which was triturated with a 1:1 mixture of ether and isopropyl ether (4 mL) and cooled overnight. Filtration gave 100mg (-4%) of white solid: mp 113-115°C; <sup>1</sup>H NMR (CDCl<sub>3</sub>) δ 6.91 (s, 2H, CH), 5.79 (s, 2H, CH<sub>2</sub>), 2.19 (s, 6H, CH<sub>3</sub>) ppm; mass spectrum (CI, CH<sub>4</sub>) m/z 219 (M+1-CH<sub>3</sub>CO<sub>2</sub>H, 26), 147 (14), 114 (43), 103 (16), 79 (19), 61 (34), 43 (100); Anal. Calcd. for C<sub>7</sub>H<sub>10</sub>N<sub>4</sub>O<sub>8</sub>: C, 30.22; H, 3.62; N, 20.14. Found: C, 30.39; H, 3.91, N, 19.89.

Trichloroacetamidine. - Trichloroacetonitrile (7.0g) was added dropwise to about 25 mL of ammonia at -40° to -50° according to a published procedure<sup>11</sup>. The cooling bath was removed and the ammonia allowed to reflux on a cold finger (dry ice) condenser for 1 h. The ammonia was allowed to evaporate and the residue was heated and stirred with petroleum ether (bp 38-50°). A cloudy solution was decanted from residual solid, filtered, and refrigerated to give needle crystals of the amidine, 4.83g (62%), with mp 48-9°C (lit mp<sup>11</sup> 47-48°).

2-Trichloromethyl-4,5-Dihydroxy-Imidazoline (19). - A solution of 4.9g (0.030 mol) of trichloroacetamidine in 7.5 mL ethanol was added dropwise to a solution of 2.2g of 40% glyoxal solution (0.015 mol) in 5 mL of water. The addition caused a temperature rise from 20° to 33°C. One hour after the addition, the flask was refrigerated at -10°C. Collection of the crystals precipitated from solution the next day gave 0.85g (26%) of the title compound: mp 107° (dec.); IR (KBr) 3400 (br, NH, OH) and 1610 (C=N) cm<sup>-1</sup>; NMR (d<sub>6</sub> - acetone) δ 5.5 (s, C-H); mass spectrum, m/z (rel intensity) 219, 221, 223 (4.7, 4.5, 1.6 for M+1), 247, 249, 251 (0.3, 0.2, 0.2 for M+29), 259, 261, 263 (0.3, 0.4, 0.1 for M+41), 59 (100).

2-Trichloromethyl-Imidazoline-4,5-Dione Hydrochloride (21). - A solution of 1.27g (0.01 mol) of oxalyl chloride in 3 mL of carbon tetrachloride was added dropwise to 1.61g (0.01 mol) trichloroacetamidine in 5 mL carbon tetrachloride. The resultant thick mixture of precipitate and solution was heated in a 65-70°C bath for 3 h. The solid was isolated by filtration, washed with carbon tetrachloride, and dried under vacuum at 20°C to give 1.73g (75%) of the title compound with mp 200°-203°C (lit<sup>12</sup> mp 194°C).

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